16. *Formal Programs for Providing NIST-Traceable Reference Materials from Commercial Sources*

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Objective: To establish formal programs to facilitate the commercial production and distribution of reference materials with a well-defined (and NIST recognized) traceability linkage to NIST.

Problem: Increased requirements for quality systems documentation for trade and effective decision-making regarding the health and safety of the U.S. population have increased the need for demonstrating "traceability-to-NIST" and establishing a more formal means for documenting measurement comparability with standards laboratories of other nations and/or regions. Standard Reference Materials (SRMs) are certified reference materials issued under NIST trademark that are well-characterized using state-of-the-art measurement methods and/or technologies for chemical composition and/or physical properties. Traditionally, SRMs have been the primary tools that NIST provided to the user community for achieving chemical measurement quality assurance and traceability to national standards. Currently, NIST provides nearly 1400 different types of SRMs and in FY98 sold nearly 37,000 SRM units to approximately 5,000 unique customers; approximately 21,000 units of these represent about 850 different types that are certified for chemical composition. NIST does not have the resources necessary to--by itself--provide the increasing quantities and specific varieties of certified reference materials (exact sample types, unique compound combinations, concentrations, etc.) needed to meet increasing needs.

Approach: The NIST Traceable Reference Materials (NTRM) program has been created as part of the solution to this problem. An NTRM is a commercially produced reference material with a well-defined traceability linkage to existing NIST standards for chemical measurements. This traceability linkage is established via criteria and protocols defined by NIST and tailored to meet the needs of the metrological community to be served. Initially the NTRM concept was implemented in the gas standards area to allow NIST to respond to increasing demands for high quality reference materials needed to implement the "Emissions Trading" provisions of the Clean Air Act of 1990 (while facing the reality of constant human and financial resources at NIST). The program has been highly successful and since its inception, ten specialty gas companies have worked with us to certify nearly 4200 NTRM cylinders of gas mixtures, which have been used to produce approximately 400,000 NIST-traceable gas standards for end-users.

The NTRM model for the commercial production of reference materials is being extended to other mature and high volume areas so that more of our resources can be diverted to address new and/or more difficult high priority measurement problems. For the benefit and protection of both the users and providers of these materials, NIST is in the process of trade marking the term NIST Traceable Reference Material (NTRM) in order to restrict its use to only those materials that meet NIST-defined criteria and specifications. Brief descriptions of each of the four areas where NTRM programs are in place or are being developed follow.

Gas NTRM Program *(F. Guenther and W. Dorko)*

The Gas NTRM program was defined to achieve maximum customer confidence in NTRM gas mixture products from Specialty Gas Companies (SGCs). At the time the program was defined it was thought that direct NIST involvement in the certification of the NTRM batches was desirable and necessary. Through this approach we have gained acceptance of these standards by the EPA, the automobile industry, and the stationary source measurement community. We now feel that the program can evolve to lessen the direct involvement of NIST in every NTRM batch. Over the next year we will be defining an alternative certification approach, which will allow SGCs who have demonstrated success through the production of NTRMs over a three-year period. This new approach will certify a particular SGC facility such that any gas cylinder analyzed on a NIST certified analytical system has the potential of being named an NTRM. Adequate controls will be imposed by NIST to assure quality control and traceability to NIST, however the SGC would be able to produce as many NTRMs as they require. It is hoped that this approach will enhance the availability of gas NTRMs such that they will replace gas SRMs as the primary traceability vehicle to U.S. Customers. Another eventual outcome of this new approach would be the worldwide acceptance of gas NTRMs.

Optical Filters NTRM Program (*G.W. Kramer and J.C. Travis*)

NIST has produced Standard Reference Materials for calibrating the wavelength scale and verifying the absorbance accuracy of UV/visible chemical spectrophotometers for several decades. The NIST production capacity is rapidly becoming insufficient to meet the demand, and a recently developed program to leverage NIST measurement capability through the private sector is being adapted to these standards. NIST-Traceable Reference Materials (NTRMs) are produced and marketed commercially, but with the active participation of NIST in the testing and value assignment of the standards. The first NTRMs for chemical spectrophotometry will be on the market within the coming year, and will be modeled on NIST SRM 930e and NIST SRM 1930, neutral density glasses (in a cuvette-simulation format) certified at five wavelengths in the visible spectral region and spanning absorbances between 0.3 and 2.0.

The expanded uncertainties for the certified values will be kept close to those of the corresponding NIST standards by reducing the recertification period from two years to one, which will be compatible with the renewal cycle of many quality control protocols and will provide more frequent cleaning of the filters. The certifying laboratories will be accredited through the NIST-based National Voluntary Laboratory Accreditation Program (NVLAP) and will maintain periodic intercomparison measurements with the National Reference Spectrophotometer in the Analytical Chemistry Division of NIST. Other spectrophotometric NTRMs for wavelength calibration and UV absorbance verification are expected to follow.

Elemental Solutions NTRM Program *(G. Turk and M. Salit)*

A workshop was held at NIST on June 3, 1999 for major commercial producers of elemental solution standards. The components of a proposed NTRM program were presented to attendees. The key elements of this program include:

-*The introduction of a new series of SRMs.* Elemental Primary Standards, or EPS SRMs.

-*A calibration transfer method that will compare the NTRM against the EPS SRMs*. These measurements would be performed or contracted by the NTRM provider

-*Proficiency testing*. The ability of the NTRM provider to distinguish between slightly different amounts of analyte in different EPS containers has been proposed, and could be combined with the calibration function by keeping the EPS values unknown to the NTRM providers.

-*Software to perform the value assignment of the NTRM with the appropriate uncertainty based on the data of the calibration transfer method*. The software (which might be Web-based) will "unlock" the assigned value only if the proficiency test is passed.

-*Periodic assessment of NTRM lots by NIST.*

We have begun to implement important aspects of this proposal within the framework of the existing SRM Single Element Standard Solutions certification program. We have designated a few NIST Primary (NP) materials, and are preparing and using weighed aliquots of NP solutions (prototype EPS) for value assignment of the Spectrometric Solutions using specially designed high precision ICPOES methods.

Metal Alloy NTRM Development *(J. Fassett and R. Greenberg)*

The first steps in producing metal alloy NTRMs were outlined at a meeting held July 22 and 23, 1999, with the company Analytical Reference Materials International, a secondary metal alloy standards producer which has agreed to work with NIST to pioneer the NTRM concept for metals. A protocol document was prepared and discussed, and the trial process begun, with the intention to "fine tune" the protocol as the process proceeds forward. The company has submitted a proposal to produce 3 NTRM candidate materials:

- AISI Low Alloy Steel
- Chrome-Moly Steel F-11
- AISI 1030 Carbon Steel.

The benchmarks to be used to provide NIST traceability will be from the existing NIST 1700 Series of Low Alloy Steels. Analytical methods to underpin the accuracy of metals SRM and NTRM certification have been developed within the Division in support of the metals NTRM and SRM programs. A quaternary alloy (designated MP35N) has been extensively analyzed within the Division in this development/demonstration exercise. The following techniques have reported results at NIST:

- *Wavelength Dispersive XRF:* Complete elemental analysis, including 19 elements;

- *Glow Discharge OES:* Comparative measurements to the 16 elements determined by industry, including important non-metals (C, P, S, Si, and B);
- *Cold Neutron Prompt Gamma Activation Analysis:* B and C;
- *Instrumental Neutron Activation Analysis:* Majors Constituents (Co, Cr, Mo, Ni) and Mn;
- *ID-TIMS:* S;
- *High precision ICP-OES:* Major Constituents (Co, Cr, Mo, Ni); and
- *Radiochemical Neutron Activation Analysis*: (P).

We need to stress that these capabilities are for the most part new to the Division. Both XRF and GD-OES measurements were made on instruments acquired in the last year with the hope that they could support the metals program, in addition to other programs in the Division. The high precision ICP-OES work was a demonstration experiment that supports our contention that this instrumental technique has primary measurement character—high precision and controlled potential sources of systematic error—and is a candidate replacement for traditional, labor-intensive classical methods. The application of ID-TIMS (for S) and the nuclear methods represent unique capabilities at NIST and are not routinely used by industry. These techniques provide an accuracy benchmark that is also unique, underpinning method-dependent (and standards-dependent) techniques used by industry.